# Long term effectiveness of the mixture of diatomaceous earth and deltamethrin on wheat

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Abstract

A mixture of diatomaceous earth (DE) and deltamethrin (DM) was developed to control stored-grain insect pests. The formulation contains a low quantity of DE and small amounts of insecticide deltamethrin technical powder dissolved in solvent and soap.

This study was initiated in order to determine how long the mixture DE/DM will provide acceptable protection against infestation by the rice weevil, *Sitophilus oryzae* (L.), the lesser grain borer *Rhyzopertha dominica* (F.) and the red flour beetle, *Tribolium castaneum* (Herbst), when applied to hard red spring wheat (HRSW) and stored under normal storage conditions. Immediately after treatment, all three species were controlled at 100 milligram per kg or parts per million (ppm) of DE/DM mixture. At 100 ppm, DE/DM mixture provided 100% population reduction of all three species for up to 12 months with little or no progeny produced.

Keywords: Diatomaceous earth, Deltamethrin, Mixture, Wheat, Protection

## *1. Introduction*

Insects infesting grain after harvest cause economic loss to producers and the grain industry. Fewer options are available for providing long term protection of grain due to concern over pesticide residues in food, insecticide resistance and the loss or restricted use of conventional grain protectants and fumigants due to new regulations. Alternatives, such as diatomaceous earth (DE), as an integral component of an Integrated Pest Management strategy, can provide very effective extended protection (Stathers et al., 2004; Athanssiou et al., 2005). However, under certain circumstances DE requires high dosage rates that have adverse effects on bulk density (test weight) and handling properties of grain, potentially reducing the value of the commodity (Korunic, 1998; Korunic et al., 1998; Fields, 1999).

Grain protectants should be safe with low mammalian toxicity, be easy to apply with minimal residue issues in finished products, have a broad spectrum of activity towards stored-grain insects and reduce progeny production, have low adverse effects on grain handling and quality properties, and have a price that is acceptable in terms of efficacy and economic viability (FAO, 1981, 1983).

Following these principles and in order to control all stored-product insects and mites within at least 7 to 21 d after treatment, even in grain having relatively high moisture content (up to 15%), a new grain protectant was developed. This new formulation is a mixture of DE, a very low concentrations of deltamethrin technical powder (DM), the synergist PBO (piperonyl butoxide), and a safe and low toxicity solvent and emulsifier for deltamethrin. The mixture combined at least two different modes of action; desiccation and chemical toxicity. Because of this combined action, the required concentrations of DE and other substances used in these mixtures are much less than if any one component were used alone (Korunic, unpublished data). The selected substances are registered as grain protectants in many countries of the world. Deltamethrin and PBO are used at approximately 0.5 to 0.7 ppm and approximately 5 ppm, respectively, for long-term protection of stored grain (EXTOXNET, 1995). In most cases, diatomaceous earth is registered to be used at concentrations of 500 to 3,500 ppm (Subramanyam and Roesli, 2000). Due to the synergism (Korunic, unpublished data) the mixture DE and deltamethrin gave very high to complete mortality of tested stored grain insects and their progeny at application rates of 18 to 20% of the effective rates of any of the active ingredients when used alone.

This mixture also produced a synergistic effect at much lower concentrations in comparison with mixtures evaluated by Daglish (1994) and with the mixtures of insecticides, from different groups, that are currently in use. When the mixture of DE and deltamethrin is applied at the recommended concentration of 100 ppm, treated grain contains only approximately 0.1 ppm of deltamethrin and 90 ppm of DE.

The objective of this study was to determining if 100 ppm of the mixture of DE and deltamethrin can protect grain during one year of storage controlling the adults and the progeny of the rice weevil, *Sitophilus oryzae* (L.) (Coleoptera: Curculionidae), the lesser grain borer, *Rhyzopertha dominica* (F.) (Coleoptera: Bostrichidae), and the red flour beetle,*Tribolium castaneum* (Herbst) (Coleoptera: Tenebrionidae).

## *2. Materials and methods*

Three species were used for these tests: *S. oryzae*. – rice weevil (RW), *R. dominica* – lesser grain borer (LGB) and *T. castaneum* – red flour beetle (RFB). A minimum of 25 adults of mixed age and gender were used for each replicate. All insect strains had been colonised in the laboratory for at least three years.

At the beginning of the test, grain was treated with 100 ppm DE/DM mixture (100 g of DE/DM mixture per 1000 kg of grain). Bioassays were initiated 0, 120, 180, 240, 300 and 360 d after the initial treatment. Comparisons were made with grain freshly treated with the same concentrations of DE/DM mixture. Treated and untreated grain was stored under ambient conditions (21.0 ± 1.0°C, 40-60% r.h.) in 11 L plastic containers with vented bottoms and lids. Grain moisture content and grain temperature were recorded throughout the experiment. Bioassays were conducted at 30° ± 1.0°C and 70% ± 5% air relative humidity (r.h.).

The variety of hard red spring wheat (HRSW) used was Quantum, grade No. 1, obtained from Palmerston Elevators, Palmerston, ON. Grain bulk density was determined using the Ohaus 0.5 L measure and Cox funnel. Grain moisture was measured using a dielectric moisture metre (AACC method 44-11). The percentage of dockage present was determined by sieving 100 g of wheat for 45 s in a 2.00-mm aperture sieve. At the beginning of the test, 3 to 6 kg of HRSW was weighed and grain moisture content, percent dockage and test weight were determined. One group was used for the initial 0-d treatment, one group was used for "fresh" treatments, and the remaining group was used as an untreated control.

The initial treatment was performed by adding 600 mg of the formulation to 6 kg grain (100 ppm) then mixing thoroughly in a plastic bag for 5 min. Test weight of the treated group was determined before transferring all groups into vented, plastic containers. Immediately after initial treatment, 500 g of grain was removed from the container for the initial treatment and from the container containing untreated control grain. Grain moisture content was determined for the untreated control and fresh treatment samples. Freshly treated grain was prepared by mixing 50 mg of DE/DM mixture with 500 g of untreated wheat (100 ppm) in a glass jar for 1 min and evenly divided into five 500 mL jars (replicates) . For each additional treatment at 120, 180, 240, 300 and 360 d after the beginning of the experiment, grain was also evenly divided into five replicates. After introducing 25-50 adults of each species to each jar, jars were maintained at 30° ± 1.0°C and 70% ± 5% r.h.

To determine mortality in each treatment, grain was sieved 7 and 14 d after insects were introduced, and the number of dead and living insects was recorded. All dead insects were removed 7 d post-introduction and all dead and live insects were removed after 14 d post-introduction; jars were maintained for an additional 35 d (total of 49 d post-introduction) before being sieved again to determine the number of adult offspring generated. One way ANOVA, means in tables within columns, followed by the same letter, were not significantly different; n = 5; $P>0.05$.

## *3. Results*

The hard red spring wheat used in this study contained between 0.8 and 0.9% dockage (w/w), a mean bulk density between 78.75 and 78.79 kg $hL^{-1}$, and initial grain moisture content of 12.2%. Treatment with 100 ppm of DE/DM mixture reduced bulk density by 2.82 kg $hL^{-1}$. The mean grain temperature and grain moisture content, as measured in the untreated grain over 300 d of storage, were 21.2 ± 1.0°C and

12.3 ± 0.3%, respectively. The low moisture content of grain and very low percentage of dockage are the probable reasons for very low development of the red flour beetle progeny.

Mortality of *S. oryzae*, *R. dominica* and *T. castaneum* was 100% on grain stored for 360 d post-treatment. The same results were achieved on grain freshly treated with DE/DM mixture (Tables 1, 2 and 3). Also, no live progeny were observed on treated grain for 360 d post-treatment. The only exception was *S. oryzae* with 99.6% progeny reduction (Table 1, 2 and 3).

**Table 1** *Sitophilus oryzae* mortality on DE/DM mixture treated grain, immediately after treatment (zero day), 180 and 360 days post-treatment.

| Treatment | Conc. (ppm) | Mean percent *S. oryzae* mortality ± s.d. after days 7 | 14 | Mean number of live progeny ± s.d. |
|---|---|---|---|---|
| Zero day-post-treatment | | | | |
| Untreated | 0 | 0.8 ± 1.8 b | - | 410.4 ± 161.2 a |
| DE/DM mixture | 100 | 100.0 ± 0.0 a | - | 0.0 ± 0.0 b |
| 180 days post-treatment | | | | |
| Untreated | 0 | 0.0 ± 0.0 a | 0.8 ± 1.8 a | 107.2 ± 20.0 a |
| DE/DM 180 days post- treatment | 100 | 87.2 ± 3.3 b | 100.0 ± 0.0 b | 0.0 ± 0.0 b |
| DE/DM fresh treatment | 100 | 84.8 ± 3.3 b | 100.0 ± 0.0 b | 0.0 ± 0.0 b |
| 360 days post-treatment | | | | |
| Untreated | 0 | 0.8 ± 1.7 a | 3.2± 1.7 **a** | 54.4 ± 32.2 a |
| DE/DM 360 days post- treatment | 100 | 97.2 ± 2.8 b | 100.0 ± 0.0 b | 0.0 ± 0.0 b |
| DE/DM fresh treatment | 100 | 100.0 ± 0.0 b | - | 0.0 ± 0.0 b |

**Table 2** *Rhyzopertha dominica* mortality on DE/DM mixture treated grain, immediately after treatment (zero day), 180 and 360 days post-treatment.

| Treatment | Conc. (ppm) | Mean percent *R. dominica* mortality ± s.d. after days 7 | 14 | Mean number of live progeny ± s.d. |
|---|---|---|---|---|
| Zero day-post-treatment | | | | |
| Untreated | 0 | 1.2 ± 1.1 a | 2.0 ± 2.0 a | 224.6 ± 44.1 a |
| DE/DM mixture | 100 | 89.2 ± 5.0 b | 100.0 ± 0.0 b | 0.0 ± 0.0 b |
| 180 days post-treatment | | | | |
| Untreated | 0 | 0.0 ± 0.0 a | 0.8 ± 1.8 a | 107.2 ± 20.0 a |
| DE/DM old treatment | 100 | 87.2 ± 3.3 b | 100.0 ± 0.0 b | 0.0 ± 0.0 b |
| DE/DM fresh treatment | 100 | 84.8 ± 3.3 b | 100.0 ± 0.0 b | 0.0 ± 0.0 b |
| 360 days post-treatment | | | | |
| Untreated | 0 | 0.0± 0.0 a | 0.0± 0.0 a | 157.8± 80.2 a |
| DE/DM old treatment | 100 | 62.4± 8.7 b | 100.0± 0.0 b | 0.0± 0.0 b |
| DE/DM fresh treatment | 100 | 87.2± 7.6 b | 100.0± 0.0 b | 0.0± 0.0 b |

Our results demonstrate that, under the conditions of grain storage (21°C and 12.2% m.c.), treatment of HRSW with 100 ppm of the mixture DE/DM provides effective protection against the adults and the progeny of three tested species for at least 12 months of storage.

*4. Discussion*

Deltamethrin is very stable on grain and shows little or no tendency to penetrate individual kernels, therefore, it is expected to be removed with the bran during processing. Results from a number of tests to determine the persistence of deltamethrin residue on stored wheat and maize were analyzed by Food and Agricultural Organization/World Health Organization (FAO/WHO, 1981, 1983). It is clear from this review that there was little or no degradation after 30 to 50 wks of storage when good storage practices were followed. For example, at 25°C, the half lives of deltamethrin on wheat at 12% m.c. and 15% m.c. were 114 and 90 wks, respectively. At 35°C, the half lives of deltamethrin on wheat at 12% m.c. and

15% m.c. were 70 and 35 wks, respectively. Degradation is faster at higher temperatures and grain moisture contents. The data for chemical residue levels and biological activity clearly indicate that deltamethrin should give prolonged residual action against grain insects. However, as some decay of the compound will occur during typical storage periods, the initial application rates will need to be at or below the maximum residue limits.

**Table 3** *Tribolium castaneum* mortality on DE/DM mixture treated grain, immediately after treatment (zero day), 180 and 360 days post-treatment.

| Treatment | Conc. (ppm) | Mean percent *T. castaneum* mortality ± s.d. after days 7 | 14 | Mean number of live progeny ± s.d. |
|---|---|---|---|---|
| Zero day-post-treatment | | | | |
| Untreated | 0 | 14.4 ± 13.1 b | 29.6 ± 17.3 b | 0.8 ± 1.1 a |
| DE/DM mixture | 100 | 95.2 ± 1.8 a | 100.0 ± 0.0 a | 0.0 ± 0.0 a |
| 180 days post-treatment | | | | |
| Untreated | 0 | 0.0 ± 0.0 a | 2.4 ± 5.4 a | 17.0 ± 10.6 a |
| DE/DM old treatment | 100 | 58.8 ± 10.0 b | 100.0 ± 0.0 b | 0.0 ± 0.0 b |
| DE/DM fresh treatment | 100 | 68.0 ± 7.0 b | 100.0 ± 0.0 b | 0.0 ± 0.0 b |
| 360 days post-treatment | | | | |
| Untreated | 0 | 0.8± 1.7 a | 9.6 ± 4.5 a | 0.6 ± 1.3 a |
| DE/DM old treatment | 100 | 68.0 ± 7.0 b | 100.0 ± 0.0 b | 0.0 ± 0.0 a |
| DE/DM fresh treatment | 100 | 72.6 ± 3.0 b | 100.0± 0.0 b | 0.0 ± 0.0 a |

There is no degradation of deltamethrin when treated wheat is milled to produce either whole wheat flour or white flour. Generally, there is a reduction in the deltamethrin level to about 10 to 20% of the level applied to wheat when it is used in the production of white flour and bread. Residues of deltamethrin are not significantly reduced during baking (FAO/WHO, 1981, 1983).

The FAO/WHO (1983) recommended the following maximum residue limits in cereal grains and milled cereal products: cereal grains 2 mg kg$^{-1}$ (2 ppm), raw wheat bran 5 mg kg$^{-1}$, whole wheat flour, wheat flour white 0.5 mg kg$^{-1}$. When DE/DM mixture formulation is applied at the recommended concentration of 100 ppm, it contains 0.01 ppm which is only 4.95% of the maximum residue limit in cereal grains recommended by FAO/WHO (1983).

Pyrethroids synergized with piperonyl butoxide, applied in a combination with organophosphates (OP), have been used in control programs in Australia, the United Kingdom, and other European countries for several years. Using a mixture of compounds from different insecticide groups seems to be a logical and economical approach. Unlike OP, pyrethroids do not rapidly break down at high temperatures and moisture contents. However, the concentrations of pyrethroids that control lesser grain borer do not completely control weevils and flour beetles. Similarly, the concentrations of OP that control many pest species do not completely control lesser grain borer.

One of the potentially valuable uses of DE is in an admixture where DE is impregnated with an insecticide. Effective insecticide concentrations can be significantly reduced in this form, and the insecticides are more readily removed from the grain (Desmarchelier, personal communication). Desmarchelier stated it was possible to halve the applied dose of insecticides such as chlorpyrifos methyl and deltamethrin by simultaneous application of small amount (100 g t$^{-1}$) of diatomaceous earth, without a reduction in efficacy.

According to the study of Arthur and Zehner (1994), the rates of degradation of the active ingredients in the mixtures of OP and pyrethroids are similar to their rates of degradation when they are applied alone. They evaluated the long term efficacy of deltamethrin applied to wheat at rates of 0.5, 0.75, and 1.0 ppm. Treated wheat was stored under ambient conditions for 10 months. Bioassays with *R. dominica* and *S. oryzae* were initiated every two months. *R. dominica* adult survival after the initial exposure was variable at all three concentrations tested throughout the 10 month period. However, no progeny were produced and the wheat was not damaged. Also, a significant number of *S. oryzae* adults survived on wheat treated with 0.5 and 0.75 ppm. Survival decreased as the application rates increased. Survival rates were 1 to 3% at 1.0 ppm, and 92 to 97% on untreated wheat. Also, subsequent progeny production decreased with increasing rates of deltamethrin (1 to 7 adults at 1.0 ppm compared with 1198 to 1662 adults on untreated wheat).

When compared with mentioned results of the research, it is clear that the main advantage of using the DE/DM mixture is acceptable efficacy in grain treated with very low doses of deltamethrin (0.1 ppm) and DE (90 ppm). These doses are much lower in comparison with the doses required for effective control when these two insecticides are applied either alone or in previously evaluated mixtures. Deltamethrin and PBO are used at approximately 0.5 to 0.75 ppm and approximately 5 ppm, respectively, for the long-term protection of stored grain. In most cases, diatomaceous earth is registered to be used at concentrations of 500 to 3500 ppm (Korunic, 1998; Subramanyam and Roesli, 2000).

Confirmed synergism between the components in DE/DM mixture reduces the dosage rates of each required for effective control, significantly reducing the risk of harmful residues and adverse effects on grain bulk density.

The effectiveness of DE/DM mixture on wheat after 360 d after treatment and the effectiveness on freshly treated wheat for rice weevil, lesser grain borer and red flour beetle were not significantly different. The results demonstrate that, under the conditions of grain storage (30°C and 14% m.c.), treatment of wheat with 100 ppm of DE/DM mixture provides effective protection against the adults and progeny of *S. oryzae*, *R. dominica* and *T. castaneum* for at least 12 months of storage.

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